

CLAIMS

The invention claimed is:

1. A copper-comprising sputtering target comprising:
at least 99.99 % copper, by weight;
an average grain size of from at least 1 micron to less than or
equal to about 50 microns; and
the target having a yield strength of greater than or equal to about
15 ksi.
2. The target of claim 1 wherein the target has a hardness of at least
40 HB.
3. The target of claim 1 having an ultimate tensile strength at least
15% greater than a target having a substantially identical elemental composition
with an average grain size of 50 microns.
4. The sputtering target of claim 1 wherein the hardness is at least
15% greater than a target having a substantially identical elemental composition
with an average grain size of 50 microns.

5. The sputtering target of claim 1 wherein the yield strength is at least 10% greater than a target having a substantially identical elemental composition with an average grain size of 50 microns.

6. The sputtering target of claim 1 wherein the target is monolithic and has a sputtering lifetime at least 30% longer than an alternative bonded target having a substantially identical elemental composition with an average grain size of 50 microns.

7. The target of claim 1 wherein the target comprises at least 99.999% copper, by weight

8. The target of claim 1 wherein the target comprises at least 99.9999% copper, by weight.

9. The target of claim 1 wherein the target comprises at least 99.9995% copper, by weight.

10. The target of claim 1 wherein the target comprises a grain size uniformity having a non-uniformity of less than or equal to 20%.

11. The target of claim 10 wherein the non-uniformity is less than 15% (1-sigma).

12. The target of claim 1 wherein the target comprises a grain size uniformity having a standard deviation of less than or equal to 10% (1-sigma).

13. The target of claim 1 wherein the target is diffusion bonded to a backing plate, the diffusion bond having a bond yield strength of greater than 10 ksi.

14. The target of claim 13 wherein the bond yield strength is greater than or equal to about 15 ksi.

15. The target of claim 1 wherein the average grain size is from about 5 microns to about 20 microns.

16. A copper alloy sputtering target consisting essentially of:
less than or equal to about 99.99% copper, by weight;
at least one alloying element selected from the group consisting of
Cd, Ca, Au, Ag, Be, Li, Mg, Al, Pd, Hg, Ni, In, Zn, B, Ga, Mn, Sn, Ge, W, Cr, O,
Sb, Ir, P, As, Co, Te, Fe, S, Ti, Zr, Sc, Si, Pt, Nb, Re, Mo, and Hf, a total amount
of the at least one alloying element present in the target being at least 100 ppm
and less than 10% by weight; the target having a hardness of at least 40 HB.
17. The target of claim 16 having an average grain size of less than 1
micron.
18. The target of claim 17 having a grain size uniformity standard
deviation throughout the target of less than or equal to about 15% (1-sigma).
19. The target of claim 17 having a grain size uniformity standard
deviation throughout the target of less than or equal to about 10% (1-sigma).
20. The target of claim 16 having a hardness uniformity standard
deviation of less than about 5% (1-sigma) throughout the target.
21. The target of claim 20 wherein the hardness uniformity standard
deviation is less than about 3.5% (1-sigma).

22. The target of claim 16 wherein the target is monolithic.

23. The target of claim 16 is diffusion bonded to a backing plate, the diffusion bond having a bond yield strength of greater than about 15 ksi.

24. The target of claim 16 having an orientation distribution function (ODF) of less than about 15 times random.

25. The target of claim 16 having an orientation distribution function (ODF) of less than about 5 times random.

26. The target of claim 16 having a primary grain orientation other than (220).

27. The target of claim 16 wherein the at least one alloying element is selected from the group consisting of Ag, Al, In, Zn, B, Ga, Mg, Sn, Ge, Ti, and Zr.

28. The target of claim 16 wherein the total amount of alloying elements is from about 1000 ppm to less than about 2%.

29. A copper alloy sputtering target consisting essentially of:
less than or equal to about 99.99% copper, by weight;
at least one alloying element selected from the group consisting of
Cd, Ca, Au, Ag, Be, Li, Mg, Al, Pd, Hg, Ni, In, Zn, B, Ga, Mn, Sn, Ge, W, Cr, O,
Sb, Ir, P, As, Co, Te, Fe, S, Ti, Zr, Sc, Mo, Si, Re, Pt, Nb, and Hf, a total amount
of the at least one alloying element present in the target being at least 100 ppm
and less than 10%, by weight; the target having an average grain size of from 1
micron to about 20 micron, and having a grain size uniformity with a standard
deviation of less than about 15% (1-sigma) throughout the target.

30. The target of claim 29 wherein the grain size uniformity standard
deviation is less than about 10% (1-sigma).

31. The target of claim 29 having a hardness of at least about 40 HB.

32. The target of claim 29 having a hardness uniformity comprising a
hardness standard deviation of less than about 5% of 1-sigma throughout the
target.

33. The target of claim 29 wherein the target is monolithic.

34. The target of claim 29 is diffusion bonded to a backing plate, the diffusion bond having a bond yield strength of greater than about 15 ksi.

35. The target of claim 29 having an orientation distribution function (ODF) of less than about 15 times random.

36. The target of claim 29 having an orientation distribution function (ODF) of less than about 5 times random.

37. The target of claim 29 having a primary grain orientation other than (220).

38. The target of claim 29 wherein the at least one alloying element is selected from the group consisting of Ag, Al, In, Zn, B, Ga, Mg, Sn, Ge, Ti, and Zr.

39. The target of claim 29 wherein the total amount of alloying elements is from about 1000 ppm to less than about 2%.

40. A monolithic sputtering target consisting essentially of copper and less than or equal to 10% by weight of a total amount of alloying elements.

41. The monolithic target of claim 40 wherein the total amount of alloying elements comprise at least one element selected from the group consisting of Cd, Ca, Au, Ag, Be, Li, Mg, Al, Pd, Hg, Ni, In, Zn, B, Ga, Mn, Sn, Ge, W, Cr, O, Sb, Ir, P, As, Co, Te, Fe, S, Ti, Zr, Sc, Mo, Si, Re, Pt, Nb, and Hf.

42. The monolithic target of claim 41 wherein the at least one element is selected from the group consisting of Ag, Al, Sn, and Ti

43. The monolithic target of claim 40 wherein the total amount of alloying elements is less than or equal to about 2%, by weight.

44. The monolithic target of claim 40 comprising at least 99.99% copper, by weight.

45. The monolithic target of claim 40 wherein the target comprises a circular shape having a thickness of about 1 inch.

46. The monolithic target of claim 40 comprising an average grain size of from about 15 microns to about 50 microns.

47. A bonded sputtering target consisting essentially of copper and less than or equal to 10% by weight of a total amount of alloying elements.

48. The bonded target of claim 47 wherein the total amount of alloying elements comprises at least one element selected from the group consisting of Cd, Ca, Au, Ag, Be, Li, Mg, Al, Pd, Hg, Ni, In, Zn, B, Ga, Mn, Sn, Ge, W, Cr, O, Sb, Ir, P, As, Co, Te, Fe, S, Ti, Zr, Sc, Mo, Si, Re, Pt, Nb, and Hf.

49. The bonded target of claim 48 wherein the at least one element is selected from the group consisting Ag, Al, Sn, and Ti.

50. The bonded target of claim 47 wherein the total amount of alloying elements is less than or equal to about 2%, by weight.

51. The bonded target of claim 47 comprising at least 99.99% copper, by weight.

52. The bonded target of claim 47 wherein the target comprises a circular shape.

53. The bonded target of claim 47 comprising an average grain size of less than about 100 microns.

54. The bonded target of claim 53 wherein the average grain size is from about 15 microns to about 50 microns.

55. The bonded target of claim 53 wherein the average grain size is less than about 30 microns.

56. The bonded target of claim 47 wherein the target is diffusion bonded to a backing plate with a bond strength of at least about 15 ksi.

57. The bonded target of claim 56 wherein the bond strength is at least about 30 ksi

58. The bonded target of claim 56 wherein the backing plate is a CuCr backing plate.

59. A method of forming a monolithic sputtering target, comprising:
providing a copper billet consisting essentially of copper and less than or equal to 10% by weight of a total amount of one or more alloying elements;

heating the billet to a temperature of at least about 900°F and maintaining the temperature for at least about 45 minutes;

hot-forging the billet with a reduction in height of at least about 50% to form a forged block;

cold rolling the forged block to a reduction of at least about 60% to form a blank;

heating the blank to induce recrystallization and form a final grain distribution having an average grain size less than about 100 microns; and

forming the blank into a monolithic target shape.

60. The method of claim 59 further comprising water quenching after the hot-forging.

61. The method of claim 59 wherein the one or more alloying elements comprise one or more elements selected from the group consisting of Cd, Ca, Au, Ag, Be, Li, Mg, Al, Pd, Hg, Ni, In, Zn, B, Ga, Mn, Sn, Ge, W, Cr, O, Sb, Ir, P, As, Co, Te, Fe, S, Ti, Zr, Sc, Mo, Si, Re, Pt, Nb, and Hf.

62. The method of claim 61 wherein the one or more alloying elements are selected from the group consisting of Ag, Al, Sn, and Ti.

63. A method of forming a monolithic sputtering target, comprising:
providing a copper billet comprising at least 99.99% copper, by weight;
heating the billet to a temperature of at least about 900°F and maintaining the temperature for at least about 45 minutes;
hot-forging the billet with a reduction in height of at least about 50% to form a forged block;
cold rolling the forged block to a reduction of at least about 60% to form a blank;
heating the blank to induce recrystallization and form a final grain distribution having an average grain size less than about 100 microns; and
forming the blank into a monolithic target shape.

64. The method of claim 63 wherein the average grain size of less than or equal to about 50 microns.

65. The method of claim 57 wherein the average grain size is less than or equal to about 15 microns.

66. The method of claim 57 further comprising performing equal channel angular extrusion prior to the cold rolling.

67. A method of forming a bonded sputtering target, comprising:
providing a copper billet comprising at least 99.99% copper, by weight;

heating the billet to a temperature of at least about 900°F and maintaining the temperature for at least about 45 minutes;

hot-forging the billet with a reduction in height of at least about 50% to form a forged block;

cold rolling the forged block to a reduction of at least about 50% to form a blank; and

bonding the blank to a backing plate.

68. The method of claim 67 wherein the bonding is conducted at a temperature which induces recrystallization and forms a final grain distribution having an average grain size less than about 100 microns.

69. The method of claim 67 wherein the bonding produces a bond having a strength of at least 15 ksi.

70. The method of claim 67 wherein the hot-forging comprises:
- an initial heating;
 - a partial height reduction; and
 - at least one re-heating and additional height reduction.

71. The method of claim 67, further comprising water quenching after the hot forging.

72. A method of forming a bonded sputtering target, comprising:
- providing a copper billet consisting essentially of copper and less than or equal to 10% by weight of a total amount of one or more alloying elements;
 - heating the billet to a temperature of at least about 900°F and maintaining the temperature for at least about 45 minutes;
 - hot-forging the billet with a reduction in height of at least about 50% to form a forged block;
 - cold rolling the forged block to a reduction of at least about 50% to form a blank;
 - and
 - bonding the blank to a backing plate.

73. The method of claim 72 wherein the bonding is conducted at a temperature which induces recrystallization and forms a final grain distribution having an average grain size less than about 100 microns

74. The method of claim 72 wherein the one or more alloying elements comprise one or more elements selected from the group consisting of Cd, Ca, Au, Ag, Be, Li, Mg, Al, Pd, Hg, Ni, In, Zn, B, Ga, Mn, Sn, Ge, W, Cr, O, Sb, Ir, P, As, Co, Te, Fe, S, Ti, Zr, Sc, Mo, Si, Re, Pt, Nb, and Hf.

75. The method of claim 74 wherein the one or more elements are selected from the group consisting of Ag, Al, Sn, and Ti.

76. The method of claim 72, wherein the bonding comprises diffusion bonding to produce a bond having a strength of at least 15 ksi.

77. The method of claim 72, wherein the hot-forging comprises:
an initial heating;
a partial height reduction; and
at least one re-heating and additional height reduction.

78. The method of claim 72 further comprising performing equal channel angular extrusion prior to the cold rolling.

79. A method of forming a copper-comprising sputtering target, comprising:

- providing a Cu billet having a purity of at least 99.99% copper;
- hot-forging the Cu billet at a temperature of greater than 300°C with a reduction in height of at least about 40% to form a forged block;
- water quenching the forged block;
- performing an extrusion process comprising:
 - at least four passes of the forged block through equal channel angular extrusion (ECAE); and
 - a heat-treatment comprising one or both of intermediate annealing between at least some of the at least four passes; and heating ECAE die to a temperature of from about 125°C to about 225°C during the extrusion process;
- after extrusion process, cold-rolling to a reduction of less than 90% to form a blank; and
- forming the blank into a target.

80. The method of claim 79 further comprising solutionizing the forged block prior to water quenching, the solutionizing comprising heating the forged block to a temperature of at least about 500°C and maintaining the temperature for at least about 60 minutes.

81. The method of claim 79 wherein the extrusion process comprises intermediate annealing at a temperature of from about 125°C to about 225°C for greater than about 1 hour.

82. The method of claim 79 further comprising heating the blank to recrystallize the copper and form a final grain distribution within the blank, the final grain distribution having an average grain size of from about 1 to about 20 microns; wherein the forming the blank into a target forms a monolithic target.

83. The method of claim 79 wherein the forming the blank into a target comprises forming a bonded target.

84. The method of claim 83 wherein the forming the bonded target comprises bonding the target to a backing plate, the bonding being conducted at a temperature of less than or equal to about 325°C for a time of less than about 4 hours, the bonding comprising at least one of hiping, roll cladding, soldering and diffusion bonding.

85. The method of claim 84 wherein the bonding comprises diffusion bonding to form a bond having a bond yield strength of from at least about 10 ksi to about 15 ksi.

86. The method of claim 79 wherein the average grain size is from 1 micron to about 50 microns.

87. The method of claim 86 wherein the average grain size is from 5 microns to about 20 microns.

88. The method of claim 79 wherein a uniform grain size distribution exists throughout an entirety of the blank, the uniform grain size having a standard deviation of less than 15% (1-sigma).

89. The method of claim 88 wherein the grain size uniformity standard deviation is less than about 10% (1-sigma).

90. The method of claim 79 wherein the Cu billet has a purity of at least about 99.999% copper.

91. The method of claim 79 wherein the Cu billet has a purity of at least about 99.9999% copper.

92. The method of claim 79 wherein the Cu billet has a purity of at least about 99.99995% copper.

93. The method of claim 79 wherein the at least four passes consists of from four to six passes.

94. A method of forming a copper alloy sputtering target, comprising:
providing a Cu billet consisting essentially of less than 99.99% copper and at least one alloying element selected from the group consisting of Cd, Ca, Au, Ag, Be, Li, Mg, Al, Pd, Hg, Ni, In, Zn, B, Ga, Mn, Sn, Ge, W, Cr, O, Sb, Ir, P, As, Co, Te, Fe, S, Ti, Zr, Sc, and Hf, a total amount of the at least one alloying element present in the Cu billet being at least 100 ppm and less than 10% by weight;

hot-forging the Cu billet at a temperature of greater than 300°C with a reduction in height of at least about 40% to form a forged block;

performing an extrusion process comprising:

at least four passes of the forged block through equal channel angular extrusion (ECAE); and

a heat-treatment comprising one or both of heating ECAE die during the extrusion process, and intermediate annealing at a temperature of from about 120°C to about 325°C, for a time of at least 1 hour between at least some of the at least four passes; after the extrusion process, cold-rolling to a reduction of less than about 90% to form a blank; and

forming the blank into a target.

95. The method of claim 94 wherein the extrusion process comprises heating the ECAE die to a temperature of from about 125°C to about 325°C.

96. The method of claim 94 further comprising solutionizing the forged block by heating to a temperature of at least about 500°C and maintaining the temperature for at least about 60 minutes prior to the extrusion process.

97. The method of claim 94 wherein the at least four passes consists of from four to six passes.

98. The method of claim 94 wherein during and after the extruding process the method utilizes only temperatures less than or equal to 350°C, and wherein the forming the blank into a target comprises forming a monolithic target.

99. The method of claim 94 wherein the forming the blank into a target comprises forming a bonded target.

100. The method of claim 99 further comprising performing a full static recrystallization treatment conducted at a temperature of from about 250°C to about 500°C for a time of from about 1 hour to about 8 hours, prior to the forming the bonded target.

101. The method of claim 99 further comprising performing a full static recrystallization treatment conducted at a temperature of from about 250°C to about 500°C for a time of from about 1 hour to about 8 hours after the forming the bonded target.

102. The method of claim 99 wherein the forming the bonded target comprises bonding the target to a backing plate, the bonding being conducted at a temperature of less than or equal to about 500°C for a time of less than or equal to about 4 hours, the bonding comprising at least one of hiping, roll cladding, soldering, explosive bonding, frictionless forging and diffusion bonding.

103. The method of claim 99 wherein the bonding comprises diffusion bonding to form a bond having a bond yield strength of from at least about 10 ksi to about 15 ksi.

104. The method of claim 94 wherein the average grain size is from 1 micron to about 20 microns.

105. The method of claim 104 wherein the average grain size is from about 5 microns to about 10 microns.

106. The method of claim 94 wherein the average grain size is less than 1 micron.

107. The method of claim 94 wherein a uniform grain size distribution exists throughout an entirety of the blank, the uniform grain size having a standard deviation of less than 15% (1-sigma).

108. The method of claim 107 wherein the grain size uniformity standard deviation is less than about 10% (1-sigma).

109. The method of claim 94 further comprising, prior to the extruding process, performing an aging treatment at a temperature of less than about 500°C to form precipitates having an average precipitate size of less than or equal to about 0.5 micron.